

PATENT ABSTRACTS OF JAPAN

(11)Publication number : 2000-218165

(43)Date of publication of application : 08.08.2000

(51)Int.Cl.

B01J 27/224

B01D 53/86

B01J 37/14

C04B 41/87

(21)Application number : 11-022958

(71)Applicant : IBIDEN CO LTD

(22)Date of filing : 29.01.1999

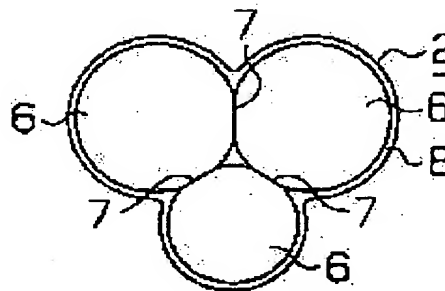
(72)Inventor : NINOMIYA TAKESHI

(54) HONEYCOMB FILTER AND ITS PREPARATION

(57)Abstract:

PROBLEM TO BE SOLVED: To provide a honeycomb filter whose breaking strength can be improved.

SOLUTION: In this honeycomb filter, a silica film 8 for increasing strength is formed in the inner face of a pore part of a base material 2 consisting of a porous silicon carbide sintered body and oxygen concn. of the base material contg. the silica film 8 is 1-10 wt.%. Oxygen in the porous silicon carbide sintered body contg. the silica film 8 exists on the surface layer part of the silicon carbide particles. It is possible thereby to improve breaking strength of the honeycomb filter.



LEGAL STATUS

[Date of request for examination]

08.04.2005

[Date of sending the examiner's decision of rejection]

[Kind of final disposal of application other than the examiner's decision of rejection or application converted registration]

[Date of final disposal for application]

[Patent number]

[Date of registration]

[Number of appeal against examiner's decision of rejection]

[Date of requesting appeal against examiner's decision of rejection]

BEST AVAILABLE COPY

[Date of extinction of right]

Copyright (C); 1998,2003 Japan Patent Office

(19) 日本国特許庁 (J P)

(12) 公開特許公報 (A)

(11) 特許出願公開番号

特開2000-218165

(P2000-218165A)

(43) 公開日 平成12年8月8日(2000.8.8)

(51) Int.Cl. ⁷	識別記号	F I	テリト [*] (参考)
B 0 1 J 27/224		B 0 1 J 27/224	A 4 D 0 4 8
B 0 1 D 53/86		37/14	4 G 0 6 9
B 0 1 J 37/14		C 0 4 B 41/87	M
C 0 4 B 41/87		B 0 1 D 53/36	C

審査請求 未請求 請求項の数3 O L (全 6 頁)

(21) 出願番号 特願平11-22958

(22) 出願日 平成11年1月29日(1999.1.29)

(71) 出願人 000000158

イビデン株式会社

岐阜県大垣市神田町2丁目1番地

(72) 発明者 二宮 健

岐阜県揖斐郡揖斐川町北方1の1 イビデ
ン 株式会社大垣北工場内

(74) 代理人 100068755

弁理士 鳳田 博宣

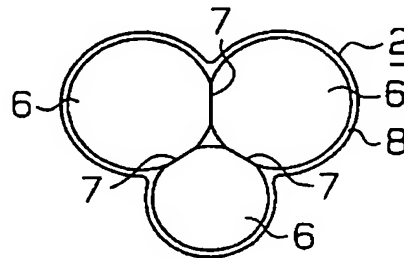
最終頁に続く

(54) 【発明の名称】 ハニカムフィルタ及びその製造方法

(57) 【要約】

【課題】破壊強度を向上することができるハニカムフィルタを提供する。

【解決手段】多孔質炭化珪素焼結体よりなる基材2は孔部内面に強度増加用のシリカ膜8が形成され、そのシリカ膜8を含む基材2の酸素濃度は1wt%~10wt%である。シリカ膜8を含む多孔質炭化珪素焼結体中の酸素は炭化珪素粒子6の表層部に存在している。



【特許請求の範囲】

【請求項1】 ハニカム状に形成された多孔質炭化珪素焼結体の孔部内面に強度増加用のシリカ膜が形成され、そのシリカ膜を含む多孔質炭化珪素焼結体の酸素濃度は1wt%～1.0wt%であるハニカムフィルタ。

【請求項2】 前記シリカ膜を含む多孔質炭化珪素焼結体中の酸素は炭化珪素粒子の表層部に存在する請求項1に記載のハニカムフィルタ。

【請求項3】 予めハニカム状に形成した多孔質炭化珪素焼結体を酸化雰囲気にて800℃～1600℃で5～100時間加熱することにより、この焼結体の孔部内面において前記焼結体の炭化珪素の一部を酸化して強度増加用のシリカ膜を形成するようにしたハニカムフィルタの製造方法。

【発明の詳細な説明】

【0001】

【発明の属する技術分野】 本発明はディーゼルエンジン等の内燃機関における排気ガスを浄化処理するために使用されるハニカムフィルタに係り、詳しくはその強度向上に関する。

【0002】

【従来の技術】 従来、例えばディーゼルエンジンの排気ガスを浄化するために、耐熱性及び熱伝導性に優れた多孔質炭化珪素焼結体をハニカム状に形成したハニカムフィルタが用いられている。このハニカムフィルタをディーゼルエンジンの排気側に接続し、このフィルタによって排気ガス中のス（カーボン）、NO_x及びHC等を酸化分解するようになっている。

【0003】 このようなハニカムフィルタの破壊強度を向上するためには、炭化珪素の焼成温度及び焼成時間を最適化することにより炭化珪素粒子同士のネック（結合部分）を成長させたり、炭化珪素粒子の粒度分布を最適化することによりネックの数を増加させたりする方法がある。

【0004】

【発明が解決しようとする課題】 しかしながら、いずれの方法を用いても多孔質炭化珪素焼結体を製造しても、多孔質炭化珪素焼結体の強度の上限があり、さらなる強度の向上を望むことはできない。また、多孔質炭化珪素焼結体よりなるハニカムフィルタには、排気ガス中のパーティキュレート（パーティクル）の濾過に適した気孔径及び気孔率が存在する。このような気孔径、気孔率を備えた多孔質炭化珪素焼結体を得るためには、炭化珪素粒子の粒度分布や炭化珪素の焼成温度及び焼成時間にも制限が加えられる。

【0005】 従って、このように製造されたハニカムフィルタの中には十分な強度がないため、捕集されたパーティキュレート中のス（カーボン）の燃焼再生時に発生する熱応力に耐え切れず、破壊してしまうものがある。

【0006】 本発明は上記の事情を考慮してなされたものであって、その目的は多孔質炭化珪素焼結体よりなる

ハニカムフィルタの強度を向上することができ、極めて簡単な方法によって多孔質炭化珪素焼結体の孔部内面に所望の厚さのシリカ膜を確実に形成することが可能なハニカムフィルタ及びその製造方法を提供することにある。

【0007】

【課題を解決するための手段】 上記の目的を達成するために、請求項1に記載の発明は、ハニカム状に形成された多孔質炭化珪素焼結体の孔部内面に強度増加用のシリカ膜が形成され、そのシリカ膜を含む多孔質炭化珪素焼結体の酸素濃度は1wt%～1.0wt%としたことを要旨とする。

【0008】 請求項2に記載の発明は、請求項1に記載のハニカムフィルタにおいて、前記シリカ膜を含む多孔質炭化珪素焼結体中の酸素は炭化珪素粒子の表層部に存在することを要旨とする。

【0009】 請求項3に記載の発明は、予めハニカム状に形成した多孔質炭化珪素焼結体を酸化雰囲気にて800℃～1600℃で5～100時間加熱することにより、この焼結体の孔部内面において前記焼結体の炭化珪素の一部を酸化して強度増加用のシリカ膜を形成するようにしたハニカムフィルタの製造方法を要旨とする。

【0010】 請求項3に記載の製造方法によってハニカムフィルタを製造すると、多孔質炭化珪素焼結体の孔部内面において、炭化珪素の一部が酸化されて、炭化珪素粒子の表層部にシリカ膜が形成される。シリカ膜を含む多孔質炭化珪素焼結体の酸素濃度は1wt%～1.0wt%の範囲であることが必要である。このシリカ膜によって炭化珪素粒子間のネックが成長するとともに、ネックの結合角度が大きくなってネックの結合端部が滑らかになり、結合強度が向上するとともに、応力集中が緩和され、ハニカムフィルタの破壊強度が向上する。

【0011】

【発明の実施の形態】 以下、本発明の一実施形態を図面に従って説明する。図1及び図2に示すように、ハニカムフィルタ1の基材2は高い融点（～3000℃）を有する多孔質炭化珪素焼結体によってハニカム状に形成されると共に、全体として四角柱状をなしている。なお、基材2は2000～2100℃の温度で焼結されている。そして、この基材2には軸線方向に平行に延びる多数のガス通過孔3が形成され、各ガス通過孔3の供給側及び排出側のいずれか一端が炭化珪素質の薄片4によって交互に封止されている。この基材2において、各ガス通過孔3の内壁面には所定の酸素濃度（1～1.0wt%）を有するシリカ膜が形成されている。

【0012】 そこで、基材2の製造方法について説明すると、まず、炭化珪素粉末を主成分とする原料により、公知の方法に従ってハニカム状基材2を焼成する。このとき、図3、4に示すように、基材2における炭化珪素粒子6同士は互いにネック7にて結合しており、炭化珪

素粒子6間のネック7の結合角度は小さくネック7の結合端部は先鋭状になっている。従って、この状態で基材2に曲げ荷重が作用すると、ネック7の結合端部に応力が集中する。

【0013】そして、この基材2を炭化珪素製の炉に入れ、炉内を空気雰囲気、すなわち酸化雰囲気とするとともに、炉内の温度を5～100時間にわたって800～1600℃の範囲の温度に保持する。

【0014】この加熱温度が800℃未満であると、酸化反応が起こり難く、1600℃を越えると、酸化反応が進みすぎて、シリカ膜が焼結体内部まで形成されて、強度低下を招く。

【0015】上記の加熱処理により、図5、6に示すように、各ガス通過孔3の内壁面及び基材2の表面において、炭化珪素粒子6の表層部が酸化され、所定の酸素濃度(1～10wt%)のシリカ膜8が形成される。このシリカ膜8によって炭化珪素粒子6間のネック7が成長するとともに、ネック7の結合角度が大きくなってネック7の結合端部が滑らかになる。従って、ネック7の結合強度が向上するとともに、ネック7の結合端部での応力集中が緩和され、基材2の破壊強度が向上する。このシリカ膜の生成量は前記空気量、加熱時間、加熱温度等に依存して変化するため、これらを制御することにより、所望の酸素濃度の均一なシリカ膜8を形成できる。

【0016】その後、前記シリカ膜8に、白金に代表される白金族元素やその他の金属元素及びその氧化物等からなる酸化触媒を担持させ、ガス通過孔3の供給側及び排出側のいずれか一端を炭化珪素質の小片4によって交互に封止すれば、内燃機関等の排気ガスを浄化するためのハニカムフィルタ1が形成される。

【0017】そして、排気ガスが排気通路5内において、図1に矢印Aで示すように、その供給側からハニカムフィルタ1内に導入されると、ガス通過孔3間の壁部により、排気ガス中のススやHC等が濾過されると共に、シリカ膜8上の触媒により、燃焼再生される。そして、浄化された排気ガスが矢印Bで示すように、ハニカムフィルタ1から排出される。

【0018】

【実施例】以下、実施例により本発明をさらに具体的に説明する。

(実施例1) 炭化珪素粉末を主成分とする原料の押出成形加工により、図2において辺L1、L2がそれぞれ3mmの正方形をなし、図1において長さL3が150mmの四角柱状をなすハニカム状基材2を形成した。このハニカム基材2において、辺L1、L2に沿って正方形の開口部を有する18個のガス通過孔3を形成し、ガス通過孔3の開口部の一边Wを1.8mm、壁厚Dを0.36mmに設定した。この基材2を炭化珪素製の炉内に入れ、炉内を空気雰囲気とするとともに、1400℃で30時間にわたって加熱した。

【0019】その結果、表1に示すように、基材2の重量は126.135gから5.331g増加して131.466gとなり、ガス通過孔3の内面全体に、酸素濃度として4.23wt%のシリカ膜8が形成された。

【0020】次に、上記のようにして得られた基材2の破壊荷重をオートグラフを用いて3点曲げ法で測定した。なお、下部スパンは135mmとし、ヘッドスピードは0.5mm/分に設定した。シリカ膜8を含む基材2の破壊強度は、図7に示すように約550kg/cm²であった。ちなみに、前記基材2と同様の形状寸法及びほぼ重量を備えシリカ膜を形成していない基材の破壊強度は、図7に示すように350kg/cm²であった。

【0021】上記の基材2の圧力損失は、図8に示すように10.5KPaであった。図9に示すように、このときの気孔径は約8.5μmであり、気孔率は約44.5%であった。ちなみに、シリカ膜を形成していない基材の圧力損失は9.8KPaであり、このときの気孔径は約9μm、気孔率は約46%であった。

【0022】(実施例2) 実施例1の基材2と同様の形状寸法を備えた多孔質炭化珪素焼結体よりなる基材2を、空気雰囲気の炉内で1450℃で90時間にわたって加熱した。

【0023】その結果、表1に示すように、基材2の重量は125.121gから10.552g増加して135.673gとなり、ガス通過孔3の内面全体に、酸素濃度として8.43wt%のシリカ膜8が形成された。

【0024】次に、上記のようにして得られた基材2の破壊荷重を実施例1と同様にして測定した。この実施例のシリカ膜8を含む基材2の破壊強度は、図7に示すように約450kg/cm²であった。

【0025】上記の基材2の圧力損失は、図8に示すように12.6KPaであった。図9に示すように、このときの気孔径は約8.1μmであり、気孔率は約44%であった。

【0026】(比較例1) 実施例1の基材2と同様の形状寸法を備えた多孔質炭化珪素焼結体よりなる基材2を、空気雰囲気の炉内で1300℃で300時間にわたって加熱した。

【0027】その結果、表1に示すように、基材2の重量は125.562gから16.257g増加して141.819gとなり、ガス通過孔3の内面全体に、酸素濃度として12.94wt%のシリカ膜8が形成された。

【0028】次に、上記のようにして得られた基材2の破壊荷重を実施例1と同様にして測定した。この実施例のシリカ膜8を含む基材2の破壊強度は、図7に示すように約460kg/cm²であった。

【0029】上記の基材2の圧力損失は、図8に示すように9.9KPaであった。図9に示すように、このときの気孔径は約8.9μmであり、気孔率は約45.5%であった。

【0030】（比較例2）実施例1の基材2と同様の形状寸法を備えた多孔質炭化珪素焼結体よりなる基材2を、空気雰囲気の中で1300℃で4時間にわたって加熱した。

【0031】その結果、表1に示すように、基材2の重量は125.479gから1.002g増加して126.481gとなり、ガス通過孔3の内面全体に、酸素濃度として0.799wt%のシリカ膜が形成された。

【0032】次に、上記のようにして得られた基材2の破壊荷重を実施例1と同様にして測定した。この実施例*10

	処理前重量	処理後重量	重量増加	酸素濃度
実施例1	126.135 g	131.466 g	5.331 g	4.23 wt%
実施例2	125.121 g	135.673 g	10.552 g	8.43 wt%
比較例1	125.562 g	141.819 g	16.257 g	12.94 wt%
比較例2	125.479 g	126.481 g	1.002 g	0.799 wt%

上記の実施例1、2及び比較例1、2の破壊強度の測定結果に基づいて、図7に示すように基材2の破壊強度と酸素濃度との関係を得ることができる。基材2の酸素濃度が1～10wt%の範囲であると、破壊強度は390kq/cm²～550kq/cm²の範囲となり、シリカ膜が形成されていない基材の破壊強度の1.11～1.57倍の破壊強度を得ることができる。また、基材2の酸素濃度が2～8wt%の範囲であると、破壊強度は480kq/cm²～550kq/cm²の範囲となり、シリカ膜が形成されていない基材の破壊強度の1.37～1.57倍の破壊強度を得ることができる。さらに、基材2の酸素濃度が4～6wt%の範囲であると、破壊強度は540kq/cm²～550kq/cm²の範囲となり、シリカ膜が形成されていない基材の破壊強度の1.54～1.57倍の破壊強度を得ることができる。

【0035】また、実施例1、2及び比較例1、2の圧力損失の測定結果に基づいて図8に示すように基材2の圧力損失と酸素濃度との関係を得ることができる。さらに、実施例1、2及び比較例1、2の気孔径及び気孔率の測定結果に基づいて図9に示すように基材2の気孔径及び気孔率と酸素濃度との関係を得ることができる。図8及び図9に基づいて、基材2の気孔径及び気孔率が低下すればするほど、圧力損失が大きくなることが分かる。

【0036】以上のことから、破壊強度を向上しつつ、圧力損失が15KPa未満の基材2を得るためには、酸素濃度として1～10wt%のシリカ膜8を基材2の炭化珪素粒子の表層部に形成すればよい。また、1～10wt%のシリカ膜8は、基材2を酸化雰囲気にて800℃～1600℃で5～100時間加熱することにより得ることができる。

【0037】また、酸素濃度として2～8wt%のシリカ膜8を形成した基材2は、シリカ膜が形成されていない基材の破壊強度の1.37～1.57倍の破壊強度を得

*のシリカ膜8を含む基材2の破壊強度は、シリカ膜を形成していない基材の破壊強度350kq/cm²以下であった。

【0033】上記の基材2の圧力損失は、図8に示すように15KPaよりはるかに上回っていた。図9に示すように、このときの気孔径も8μmを下回っており、気孔率は44%を下回っていた。

【0034】

【表1】

ることができるとともに、圧力損失を12.5KPa未満とすることができ、良好な特性のハニカムフィルタを得ることができる。

【0038】さらに、酸素濃度として4～6wt%のシリカ膜8を形成した基材2は、シリカ膜が形成されていない基材の破壊強度の約1.5倍以上の破壊強度を得ることができるとともに、圧力損失を12.5KPa未満とすることができ、より良好な特性のハニカムフィルタを得ることができる。

【0039】なお、上記実施形態は次のように変更してもよく、その場合でも同様の作用および効果を得ることができる。

・ 上記実施形態では、基材2は全体として四角柱状に形成したが、円柱状に形成した基材に実施してもよい。

【0040】・ 上記実施形態では、基材2を空気雰囲気中で加熱することにより炭化珪素粒子を酸化させたが、炭化珪素粒子の酸化はこれに限定されるものではない。次に、上記実施形態から把握できる他の技術的思想を以下に記載する。

【0041】・ 請求項1及び2のいずれか1項に記載のハニカムフィルタにおいて、前記シリカ膜を含む多孔質炭化珪素焼結体の酸素濃度は2wt%～8wt%であるハニカムフィルタ。

【0042】・ 請求項1及び2のいずれか1項に記載のハニカムフィルタにおいて、前記シリカ膜を含む多孔質炭化珪素焼結体の酸素濃度は4wt%～6wt%であるハニカムフィルタ。

【0043】

【発明の効果】以上詳述したように、請求項1及び2のいずれかに記載の発明は、ハニカムフィルタの破壊強度を向上することができるという優れた効果を発揮する。

【0044】請求項3に記載の発明は、炭化珪素焼結体の強度低下を招くことなく、極めて簡単な方法によって孔部内面に所望のシリカ膜を確実に形成することができ

* 体を拡大して示す模式図。

【図7】多孔質炭化珪素焼結体の破壊強度と酸素濃度との関係を示す線図。

【図8】多孔質炭化珪素焼結体の圧力損失と酸素濃度との関係を示す線図。

【図9】多孔質炭化珪素焼結体の気孔径及び気孔率と酸素濃度との関係を示す線図。

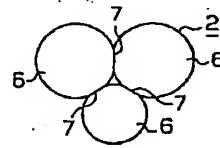
【符号の説明】

2…基材、3…ガス通過孔、6…炭化珪素粒子、7…ネック、8…シリカ膜。

10 ック、8…シリカ膜。

10 ック、8…シリカ膜。

【圖 3】

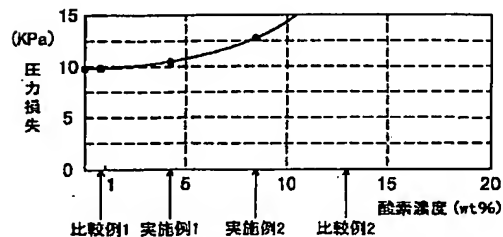


【圖7】

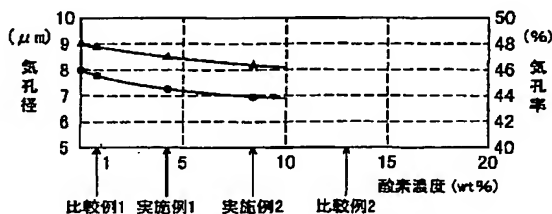


Figure 1 is a line graph showing the relationship between breaking strength (kgf/cm²) and urea concentration (wt%). The Y-axis represents breaking strength, ranging from 200 to 700 kgf/cm². The X-axis represents urea concentration, ranging from 0 to 20 wt%. Four data series are plotted: Example 1 (solid line with dots), Example 2 (dashed line with dots), Comparative Example 1 (solid line with dots), and Comparative Example 2 (dashed line with dots). Example 1 shows the highest breaking strength, peaking at approximately 580 kgf/cm² at 5 wt% urea concentration. Comparative Example 1 shows the lowest breaking strength, peaking at approximately 450 kgf/cm² at 5 wt% urea concentration. Examples 2 and Comparative Example 2 show intermediate breaking strengths, peaking at approximately 500 kgf/cm² at 5 wt% urea concentration.

Urea Concentration (wt%)	Example 1 (kgf/cm²)	Example 2 (kgf/cm²)	Comparative Example 1 (kgf/cm²)	Comparative Example 2 (kgf/cm²)
1	350	400	350	400
2	450	450	400	450
4	550	500	450	500
5	580	500	450	500
6	550	500	450	500
8	500	500	450	500
10	400	500	450	500



【図9】



フロントページの続き

Fターム(参考) 4D048 AA06 AA18 AB01 AB03 BA06X
BA06Y BA45X BA45Y BB02
4G069 AA01 AA03 AA08 BA02A
BA02B BB15A BB15B BD04A
BD04B BD05A BD05B CA03
CA07 CA10 CA13 CA15 CA18
EA19 EA27 EB17Y FA04
FC07

PATENT ABSTRACTS OF JAPAN

(11)Publication number : 2000-218165

(43)Date of publication of application : 08.08.2000

(51)Int.Cl.

B01J 27/224

B01D 53/86

B01J 37/14

C04B 41/87

(21)Application number : 11-022958

(71)Applicant : IBIDEN CO LTD

(22)Date of filing : 29.01.1999

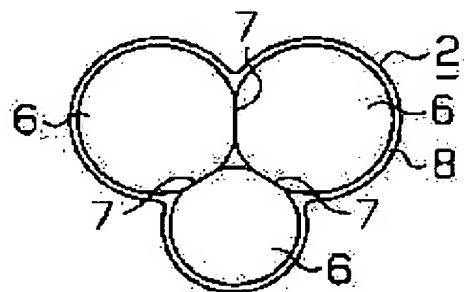
(72)Inventor : NINOMIYA TAKESHI

(54) HONEYCOMB FILTER AND ITS PREPARATION

(57)Abstract:

PROBLEM TO BE SOLVED: To provide a honeycomb filter whose breaking strength can be improved.

SOLUTION: In this honeycomb filter, a silica film 8 for increasing strength is formed in the inner face of a pore part of a base material 2 consisting of a porous silicon carbide sintered body and oxygen concn. of the base material contg. the silica film 8 is 1-10 wt.%. Oxygen in the porous silicon carbide sintered body contg. the silica film 8 exists on the surface layer part of the silicon carbide particles. It is possible thereby to improve breaking strength of the honeycomb filter.



LEGAL STATUS

[Date of request for examination]

08.04.2005

[Date of sending the examiner's decision of rejection]

[Kind of final disposal of application other than the examiner's decision of rejection or application converted registration]

[Date of final disposal for application]

[Patent number]

[Date of registration]

[Number of appeal against examiner's decision of rejection]

[Date of requesting appeal against examiner's decision of rejection]

[Date of extinction of right]

* NOTICES *

JPO and NCIPi are not responsible for any damages caused by the use of this translation.

1. This document has been translated by computer. So the translation may not reflect the original precisely.
2. **** shows the word which can not be translated.
3. In the drawings, any words are not translated.

CLAIMS

[Claim(s)]

[Claim 1] The oxygen density of the porosity silicon carbide sintered compact which the silica film for an increment on the strength is formed in the pore inside of the porosity silicon carbide sintered compact formed in the shape of a honeycomb, and contains the silica film is a honeycomb filter which is 1wt% - 10wt%.

[Claim 2] The oxygen in the porosity silicon carbide sintered compact containing said silica film is a honeycomb filter according to claim 1 which exists in the surface section of a silicon carbide particle.

[Claim 3] The manufacture approach of the honeycomb filter which oxidizes in some silicon carbide of said sintered compact in the pore inside of this sintered compact, and formed the silica film for an increment on the strength by heating the porosity silicon carbide sintered compact beforehand formed in the shape of a honeycomb at 800 degrees C - 1600 degrees C according to an oxidizing atmosphere for 5 to 100 hours.

[Translation done.]

* NOTICES *

JPO and NCIPi are not responsible for any damages caused by the use of this translation.

1. This document has been translated by computer. So the translation may not reflect the original precisely.
2. **** shows the word which can not be translated.
3. In the drawings, any words are not translated.

DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001]

[Field of the Invention] This invention relates to the honeycomb filter used in order to carry out purification processing of the exhaust gas in internal combustion engines, such as a diesel power plant, and relates to the improvement in on the strength in detail.

[0002]

[Description of the Prior Art] In order to purify the exhaust gas of the former, for example, a diesel power plant, the honeycomb filter which formed the porosity silicon carbide sintered compact excellent in thermal resistance and thermal conductivity in the shape of a honeycomb is used. This honeycomb filter is connected to the exhaust side of a diesel power plant, and oxidative degradation of the soot in exhaust gas (carbon), NOx, HC, etc. is carried out with this filter.

[0003] In order to improve the disruptive strength of such a honeycomb filter, there is the approach of increasing, and it being sufficient for and carrying out the number of necks by optimizing the burning temperature and firing time of silicon carbide by growing up the neck (a part for a bond part) of silicon carbide particles, or optimizing the particle size distribution of a silicon carbide particle.

[0004]

[Problem(s) to be Solved by the Invention] However, even if it manufactures a porosity silicon carbide sintered compact using which approach, there is an upper limit of the reinforcement of a porosity silicon carbide sintered compact, and improvement in the further reinforcement cannot be desired. Moreover, the pore diameter and porosity suitable for the particulate filtration in exhaust gas exist in the honeycomb filter which consists of a porosity silicon carbide sintered compact. In order to obtain the porosity silicon carbide sintered compact equipped with such a pore diameter and porosity, a limit is added also to the particle size distribution of a silicon carbide particle, the burning temperature of silicon carbide, and firing time.

[0005] Therefore, since there is no reinforcement sufficient in the honeycomb filter manufactured in this way, the thermal stress generated at the time of combustion playback of the soot in the particulate by which uptake was carried out is borne, and it does not go out, but there are some which are destroyed.

[0006] This invention is made in consideration of the above-mentioned situation, the purpose can improve the reinforcement of the honeycomb filter which consists of a porosity silicon carbide sintered compact, and it is in offering the honeycomb filter which can form the silica film of desired thickness in the pore inside of a porosity silicon carbide sintered compact certainly by the very easy approach, and its manufacture approach.

[0007]

[Means for Solving the Problem] In order to attain the above-mentioned purpose, let it be a summary for the silica film for an increment on the strength to have made the oxygen density of the porosity silicon carbide sintered compact which is formed and contains the silica film 1wt% - 10wt% at the pore inside of the porosity silicon carbide sintered compact with which invention according to claim 1 was formed in the shape of a honeycomb.

[0008] Let it be a summary for the oxygen in the porosity silicon carbide sintered compact with which invention according to claim 2 contains said silica film in a honeycomb filter according to claim 1 to exist in the surface section of a silicon carbide particle.

[0009] Invention according to claim 3 makes a summary the manufacture approach of the honeycomb filter which oxidizes in some silicon carbide of said sintered compact in the pore inside of this sintered compact, and formed the silica film for an increment on the strength by heating the porosity silicon carbide sintered compact beforehand formed in the shape of a honeycomb at 800 degrees C - 1600 degrees C according to an

oxidizing atmosphere for 5 to 100 hours.

[0010] If a honeycomb filter is manufactured by the manufacture approach according to claim 3, in the pore inside of a porosity silicon carbide sintered compact, some silicon carbide will oxidize and the silica film will be formed in the surface section of a silicon carbide particle. The oxygen density of the porosity silicon carbide sintered compact containing the silica film needs to be the range of 1wt% - 10wt%. While whenever [bond angle / of a neck] becomes large, the joint edge of a neck becomes smooth, while the neck between silicon carbide particles grows with this silica film, and bond strength improves, stress concentration is eased and the disruptive strength of a honeycomb filter improves.

[0011]

[Embodiment of the Invention] Hereafter, 1 operation gestalt of this invention is explained according to a drawing. As shown in drawing 1 and drawing 2, the base material 2 of a honeycomb filter 1 is making the shape of the square pole as a whole while being formed in the shape of a honeycomb with the porosity silicon carbide sintered compact which has the high melting point (- 3000 degrees C). In addition, the base material 2 is sintered at the temperature of 2000-2100 degrees C. And the gas passage hole 3 of a large number prolonged in parallel is formed in the direction of an axis at this base material 2, and the closure of the any 1 edge by the side of supply of each gas passage hole 3 and discharge is carried out by turns by the wafer 4 of the quality of silicon carbide. In this base material 2, the silica film which has a predetermined oxygen density (1 - 10wt%) is formed in the internal surface of each gas passage hole 3.

[0012] Then, explanation of the manufacture approach of a base material 2 calcinates the honeycomb-like base material 2 first with the raw material which uses silicon carbide powder as a principal component according to a well-known approach. At this time, as shown in drawing 3 and 4, silicon carbide particle 6 comrades in a base material 2 are mutually combined at the neck 7, and the joint edge of a neck 7 has become acute [-like] small [whenever / bond angle / of the neck 7 between the silicon carbide particles 6]. Therefore, if a bending load acts on a base material 2 in this condition, stress will concentrate on the joint edge of a neck 7.

[0013] And while putting this base material 2 into the furnace made from silicon carbide and making the inside of a furnace into an air ambient atmosphere, i.e., an oxidizing atmosphere, the temperature in a furnace is held to the temperature of the range of 800-1600 degrees C over 5 - 100 hours.

[0014] If oxidation reaction cannot occur easily that whenever [this stoving temperature] is less than 800 degrees C and 1600 degrees C is exceeded, oxidation reaction progresses too much, and the silica film will be formed to the interior of a sintered compact, and will cause a fall on the strength.

[0015] By the above-mentioned heat-treatment, as shown in drawing 5 and 6, in the internal surface of each gas passage hole 3, and the front face of a base material 2, the surface section of the silicon carbide particle 6 oxidizes, and the silica film 8 of a predetermined oxygen density (1 - 10wt%) is formed. While the neck 7 between the silicon carbide particles 6 grows with this silica film 8, whenever [bond angle / of a neck 7] becomes large, and the joint edge of a neck 7 becomes smooth. Therefore, while the bond strength of a neck 7 improves, the stress concentration in the joint edge of a neck 7 is eased, and the disruptive strength of a base material 2 improves. Since the amount of generation of this silica film changes depending on whenever [said air content, heating time, and stoving temperature] etc., it can form the uniform silica film 8 of a desired oxygen density by controlling these.

[0016] Then, if the oxidation catalyst which becomes said silica film 8 from the platinum group metals represented by platinum, other metallic elements, its oxide, etc. is made to support and any 1 edge by the side of supply of the gas passage hole 3 and discharge is closed by turns by the wafer 4 of the quality of silicon carbide, the honeycomb filter 1 for purifying exhaust gas, such as an internal combustion engine, will be formed.

[0017] And combustion playback is carried out by the catalyst on the silica film 8, while soot, HC, etc. in exhaust gas will be filtered by the wall between the gas passage holes 3 if introduced in a honeycomb filter 1 from the supply side as exhaust gas shows by the arrow head A in a flueway 5 at drawing 1. And as the purified exhaust gas shows by the arrow head B, it is discharged from a honeycomb filter 1.

[0018]

[Example] Hereafter, an example explains this invention still more concretely.

(Example 1) By extrusion-molding processing of the raw material which uses silicon carbide powder as a principal component, the honeycomb-like base material 2 which makes the shape of the square pole whose die length L3 is 150mm in nothing and drawing 1 about the shape of a square whose sides L1 and L2 are 33mm, respectively in drawing 2 was formed. In this honeycomb base material 2, 18 gas passage holes 3 which have square-like opening along the sides L1 and L2 were formed, one side W of opening of the gas

passage hole 3 was set as 1.8mm, and wall thickness D was set as 0.36mm. While putting in this base material 2 in the furnace made from silicon carbide and making the inside of a furnace into the air ambient atmosphere, it heated over 30 hours at 1400 degrees C.

[0019] Consequently, as shown in Table 1, the weight of a base material 2 increased 5.331g from 126.135g, and was set to 131.466g, and the 4.23wt(s)% silica film 8 was formed in the whole inside of the gas passage hole 3 as an oxygen density.

[0020] Next, the breaking load of the base material 2 obtained as mentioned above was measured by the three-point bending method using the autograph. In addition, the lower span set to 135mm and head speed was set as a part for 0.5mm/. the disruptive strength of the base material 2 containing the silica film 8 is shown in drawing 7 -- as -- about 550kg/cm² it was . incidentally, the same geometry as said base material 2 and the disruptive strength of a base material which is mostly equipped with weight and does not form the silica film are shown in drawing 7 -- as -- 350kg/cm² it was .

[0021] The pressure loss of the above-mentioned base material 2 was 10.5KPa(s) as shown in drawing 8 . As shown in drawing 9 , the pore diameter at this time was about 8.5 micrometers, and porosity was about 44.5%. Incidentally the pressure loss of the base material which does not form the silica film was 9.8KPa(s), the pore diameter at this time was about 9 micrometers, and porosity was about 46%.

[0022] (Example 2) The base material 2 of an example 1 and the base material 2 which consists of a porosity silicon carbide sintered compact equipped with the same geometry were heated over 90 hours at 1450 degrees C in the furnace of an air ambient atmosphere.

[0023] Consequently, as shown in Table 1, the weight of a base material 2 increased 10.552g from 125.121g, and was set to 135.673g, and the 8.43wt(s)% silica film was formed in the whole inside of the gas passage hole 3 as an oxygen density.

[0024] Next, the breaking load of the base material 2 obtained as mentioned above was measured like the example 1. the disruptive strength of the base material 2 containing the silica film 8 of this example is shown in drawing 7 -- as -- about 450kg/cm² it was .

[0025] The pressure loss of the above-mentioned base material 2 was 12.6KPa(s) as shown in drawing 8 . As shown in drawing 9 , the pore diameter at this time was about 8.1 micrometers, and porosity was about 44%.

[0026] (Example 1 of a comparison) The base material 2 of an example 1 and the base material 2 which consists of a porosity silicon carbide sintered compact equipped with the same geometry were heated over 300 hours at 1300 degrees C in the furnace of an air ambient atmosphere.

[0027] Consequently, as shown in Table 1, the weight of a base material 2 increased 16.257g from 125.562g, and was set to 141.819g, and the 12.94wt(s)% silica film was formed in the whole inside of the gas passage hole 3 as an oxygen density.

[0028] Next, the breaking load of the base material 2 obtained as mentioned above was measured like the example 1. the disruptive strength of the base material 2 containing the silica film 8 of this example is shown in drawing 7 -- as -- about 460kg/cm² it was .

[0029] The pressure loss of the above-mentioned base material 2 was 9.9KPa(s) as shown in drawing 8 . As shown in drawing 9 , the pore diameter at this time was about 8.9 micrometers, and porosity was about 45.5%.

[0030] (Example 2 of a comparison) The base material 2 of an example 1 and the base material 2 which consists of a porosity silicon carbide sintered compact equipped with the same geometry were heated over 4 hours at 1300 degrees C in the furnace of an air ambient atmosphere.

[0031] Consequently, as shown in Table 1, the weight of a base material 2 increased 1.002g from 125.479g, and was set to 126.481g, and the 0.799wt(s)% silica film was formed in the whole inside of the gas passage hole 3 as an oxygen density.

[0032] Next, the breaking load of the base material 2 obtained as mentioned above was measured like the example 1. The disruptive strength of the base material 2 containing the silica film 8 of this example is the disruptive strength of 350kg/cm² of the base material which does not form the silica film. It was the following.

[0033] As shown in drawing 8 , it had more easily than 15KPa(s) exceeded the pressure loss of the above-mentioned base material 2. As shown in drawing 9 , the pore diameter at this time is also less than 8 micrometers, and porosity was less than 44%.

[0034]

[Table 1]

	処理前重量	処理後重量	重量増加	酸素濃度
実施例 1	126.135 g	131.466 g	5.331 g	4.23 wt%
実施例 2	125.121 g	135.673 g	10.552 g	8.43 wt%
比較例 1	125.562 g	141.819 g	16.257 g	12.94 wt%
比較例 2	125.479 g	126.481 g	1.002 g	0.799 wt%

Based on the measurement result of the disruptive strength of the above-mentioned examples 1 and 2 and the examples 1 and 2 of a comparison, as shown in drawing 7, the relation between the disruptive strength of a base material 2 and an oxygen density can be obtained. Disruptive strength is 390kg/cm² - 550kg/cm² as the range of the oxygen density of a base material 2 is 1 - 10wt%. It becomes the range and disruptive strength 1.11 to 1.57 times the disruptive strength of the base material with which the silica film is not formed can be obtained. Moreover, disruptive strength is 480kg/cm² - 550kg/cm² as the range of the oxygen density of a base material 2 is 2 - 8wt%. It becomes the range and disruptive strength 1.37 to 1.57 times the disruptive strength of the base material with which the silica film is not formed can be obtained. Furthermore, disruptive strength is 540kg/cm² - 550kg/cm² as the range of the oxygen density of a base material 2 is 4 - 6wt%. It becomes the range and disruptive strength 1.54 to 1.57 times the disruptive strength of the base material with which the silica film is not formed can be obtained.

[0035] Moreover, as shown in drawing 8 based on the measurement result of the pressure loss of examples 1 and 2 and the examples 1 and 2 of a comparison, the relation between the pressure loss of a base material 2 and an oxygen density can be obtained. Furthermore, as shown in drawing 9 based on the pore diameter of examples 1 and 2 and the examples 1 and 2 of a comparison, and the measurement result of porosity, the relation between the pore diameter of a base material 2 and porosity, and an oxygen density can be obtained. The more the pore diameter and porosity of a base material 2 fall based on drawing 8 R> 8 and drawing 9, the more it turns out that pressure loss becomes large.

[0036] What is necessary is just to form the 1 - 10wt% silica film 8 in the surface section of the silicon carbide particle of a base material 2 as an oxygen density, in order for pressure loss to obtain the base material 2 of less than 15 KPas from the above thing, improving disruptive strength. Moreover, the 1 - 10wt% silica film 8 can be obtained by heating a base material 2 at 800 degrees C - 1600 degrees C according to an oxidizing atmosphere for 5 to 100 hours.

[0037] Moreover, the base material 2 which formed the 2 - 8wt% silica film 8 as an oxygen density can set pressure loss to less than 12.5 KPas, and can obtain the honeycomb filter of a good property while it can obtain disruptive strength 1.37 to 1.57 times the disruptive strength of the base material with which the silica film is not formed.

[0038] Furthermore, the base material 2 which formed the 4 - 6wt% silica film 8 as an oxygen density can set pressure loss to less than 12.5 KPas, and can obtain the honeycomb filter of a better property while it can obtain the disruptive strength of about 1.5 times or more of the disruptive strength of the base material with which the silica film is not formed.

[0039] In addition, the above-mentioned operation gestalt may be changed as follows, and can acquire the same operation and effectiveness even in such a case.

- With the above-mentioned operation gestalt, although the base material 2 was formed in the shape of the square pole as a whole, it may be carried out to the base material formed in the shape of a cylinder.

[0040] - Although the silicon carbide particle was oxidized by heating a base material 2 in an air ambient atmosphere with the above-mentioned operation gestalt, oxidation of a silicon carbide particle is not limited to this. Next, other technical thought which can be grasped from the above-mentioned operation gestalt is indicated below.

[0041] - The oxygen density of the porosity silicon carbide sintered compact which contains said silica film in any 1 term of claims 1 and 2 in the honeycomb filter of a publication is a honeycomb filter which is 2wt (s)% - 8wt%.

[0042] - The oxygen density of the porosity silicon carbide sintered compact which contains said silica film in any 1 term of claims 1 and 2 in the honeycomb filter of a publication is a honeycomb filter which is 4wt (s)% - 6wt%.

[0043]

[Effect of the Invention] As explained in full detail above, invention given in either of claims 1 and 2 demonstrates the outstanding effectiveness that the disruptive strength of a honeycomb filter can be improved.

[0044] Invention according to claim 3 demonstrates the outstanding effectiveness that the desired silica film

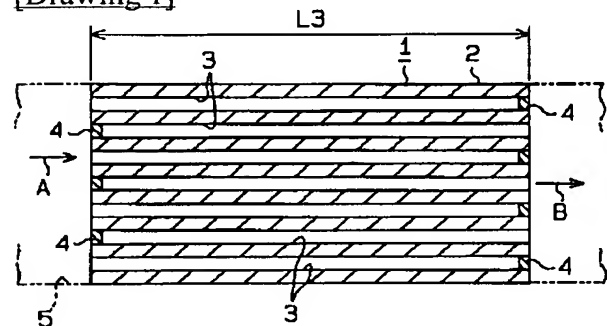
can be certainly formed in a pore inside by the very easy approach, without causing the fall of a silicon carbide sintered compact on the strength.

[Translation done.]

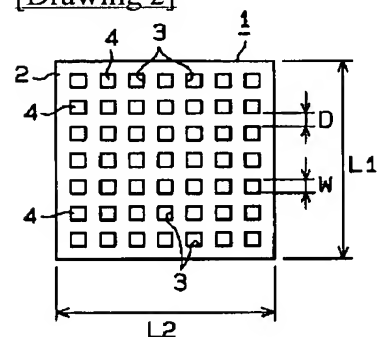
JPO and NCIPI are not responsible for any damages caused by the use of this translation.

- 1.This document has been translated by computer. So the translation may not reflect the original precisely.
- 2.**** shows the word which can not be translated.
- 3.In the drawings, any words are not translated.

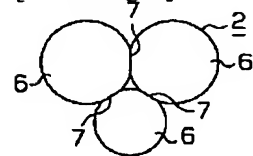
[Drawing 1]



[Drawing 2]



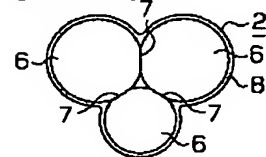
[Drawing 3]



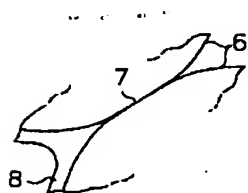
[Drawing 4]



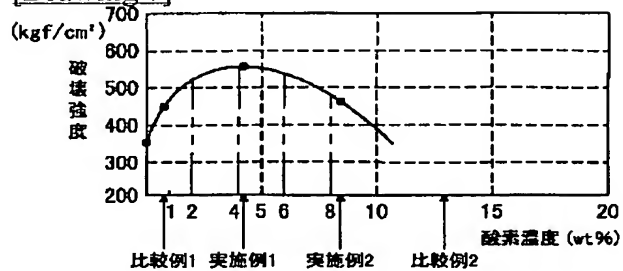
[Drawing 5]



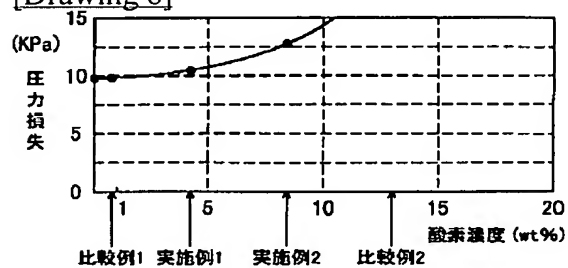
[Drawing 6]



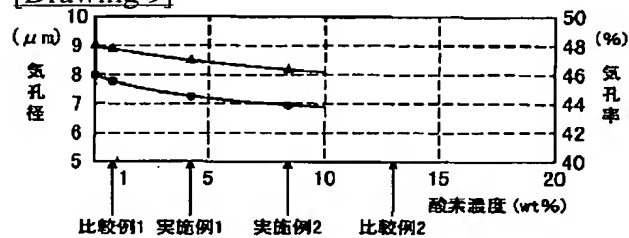
[Drawing 7]



[Drawing 8]



[Drawing 9]



[Translation done.]

**This Page is Inserted by IFW Indexing and Scanning
Operations and is not part of the Official Record**

BEST AVAILABLE IMAGES

Defective images within this document are accurate representations of the original documents submitted by the applicant.

Defects in the images include but are not limited to the items checked:

- ☐ **BLACK BORDERS**
- ☐ **IMAGE CUT OFF AT TOP, BOTTOM OR SIDES**
- ☒ **FADED TEXT OR DRAWING**
- ☒ **BLURRED OR ILLEGIBLE TEXT OR DRAWING**
- ☐ **SKEWED/SLANTED IMAGES**
- ☐ **COLOR OR BLACK AND WHITE PHOTOGRAPHS**
- ☐ **GRAY SCALE DOCUMENTS**
- ☐ **LINES OR MARKS ON ORIGINAL DOCUMENT**
- ☐ **REFERENCE(S) OR EXHIBIT(S) SUBMITTED ARE POOR QUALITY**
- ☐ **OTHER:** _____

IMAGES ARE BEST AVAILABLE COPY.

As rescanning these documents will not correct the image problems checked, please do not report these problems to the IFW Image Problem Mailbox.